Plant Operations Notebook, N-92-05-02.1

This notebook, which covers the period April 1892-February 1893, is a continuation of N-92-03-19. It was used by Edison and an unidentified author primarily for notes from experiments on reducing phosphorous. The tests are numbered 153-158. Test numbers 157 and 158 are complemented by two letters from Booth, Garrett & Blair, analytical chemists. An incomplete, inserted note by Edison regarding phosphorous tests is followed by a complete, variant version in another hand under the title, "Experiments made by Mr. Edison for the removal of Phosphorus from magnetic concentrates." The book also includes two tests for the gold properties of arsenical pyrites. The front cover is labeled "Mr. Edison's Phosphorous Expts #2." The spine is labeled "Mr. Edisons notes." The pages are unnumbered. Approximately 25 pages have been used. Thirteen loose pages have been inserted into the book.

Book 2 –

Sucrose of lead warm
[illegible] at [illegible]
~~100 cc of gms of [illegible]~~
~~Then 33 cc~~ of dil. heated 8
hours on hot plate gives
about 1/3 what Soxh. sac does
perhaps little more – [illegible]
No iron or aluminum used
~~detected~~ = whether it was
Used lead alone that detected
Cautionary [illegible] in former
Experiment took out good
deal 1/2 as much as Soxh.
but I didn't exp. how much
it was –

Oct. 5th 1000 cc [illegible]
of this No — [illegible] 33
Concentrated & then [illegible]
this [illegible] time [illegible] back
100 gm [illegible] 10 hours
on hot plate = $\frac{1}{3}$ of
what [illegible] of [illegible]
gives [illegible] & this may be
due to the stronger.
Calcium of Sucrate —
whether stronger or not
Can't say — No [illegible]
Caine out —

nothing came out —

Humic acid Residues phosCal
soluable —

following salts are to
be tried 50 grms ore
50 cc water — 1 gram of the
substance — heated on
hot plate 24 hours —

Hydrochloride Alumina
Chloride Zinc
" Cadmium
" Manganese

Iron — Cobalt, Nickel,
Copper — Lead, Mercury
Arsenic Antimony

Chloride Tin.
" Ammonium
" Potassium
" Sodium
" Lithium
" Barium
" Strontium
" Calcium
" Magnesium

Potassium - Stannochloride.
Stannic & Stannous Chloride.

Sulphates –

Sodium – Potassium,
Ammonium ~~Barium~~
Chromium, Magnesium
Zinc Ferric Sulphate
Ferrous sulphate.
Manganous & Manganic
Sulphates. Copper
Nickel Cobalt Cadmium
Alumino-Potassic Sulphate
Chromo-potassic Sulphate
Antimony Tin – Lead
~~Arsen~~ Aluminium

26 X Saracinic acid with KO — soluble soapy — whitish pp on adding Maly — dont dissolve on adding NO_3 —

All the X's are no good except Lactose & KO & Dichloroacetic acid — last rather good very little Fe & Al =

phenom — Tetraethylammon Hydrate precip Malygdic acid — white.

Acids

Succinic. Acetic,
Formic, Oxalic, Citric,
Tartaric, Valeric,
Oleic - propionic, ~~phosp~~
Boric, Phosphoric,
Tungstic, Iodic, periodic
Hydrochloric Hydrosulphuric
Hydrojodic, Sulphuric -
Hydrocyanic, Trichloroacetic
Dichloroacetic, Carbolic,
Salicylic, Nitric -
Hyposulphurous, Hydrobromic
Arsenic acid, Stannic acid
Benzoic, picric,
Borobenzoic acid

16 | 322 (20
320

20 lbs

3 | 27000 to 100000
9 00
33000

27.

30000 | 9 6 7 5 0 0 0 (327.
9 6 7 5 0 0 0

Acids Continued

Bromic, Butyric, Caproic

Chloric, perchloric

Chlorochromic, Nitrochromic

Chromic, Chrysophanic

Cresolic (Cresol)

Cyanuric acid, gallic,

Tannic, pyrogallic.

Hydrofluoric, Hydrofluosilicic

Iso-butyric, Isovaleric acid

Lactic, Malic,

Molybdic, Monobromoacetic

Monochloroacetic

Acids Continued.

Orthophenolsulphonic acid (Aseptol)

sulphoCarbolic,

Cresate,

No 153 — 500 cc

~~500~~ 500 cc water

10 gms sugar 1 gm

Soda —

used 33 cc after [illegible]

midnight [illegible] will

get warm [illegible]

[illegible] home now —

154 — ground a lot of

Ogden washed to 190

mesh (190) washed

one part to sent as 154

untreated with acid.

155 Same as 154 treated

with ~~20 cc~~ 33 cc of

a 100 cc 150 cc H_2SO_4 + heat

1 X Aethylen Monoacetat, ng
2 X Butylso-Bromat ng
3 X Methyl-propyl-Ketone ng
4 X Methyl Sulfo Cyanat ng
5 X Methyl acetat,
6 X Guajakol. Ranff - ng
7 X Nitrotoluol ng
8 X Pyrid — Hydrochlorat ng
9 X Dichloroacetic acid Very good
Got Little Fe + Al taken out
10 X Tetraethylammon Hydrate — ng — but pp Malyb dic acid or something — unsettled,
11 X Ammon Sulfocyanate. ng
12 X Ammon Benzoate ng
13 X Analin Chlorat ng
14 X acid phlalic — would not dis added KO — only part dis — ng

15 X Methylamin chlorat ng
16 X Piperonic acid KO – ng
17 X Ur-Ethane, ng
18 X phenolphtalein in KO – some but due to KO –
19 Di-Resorcin not sol added KO ng
20 – Chloral Hydrate. KO – give off chloroform ng –
20 Chloral Hydrate ng
22 Salicilin + KO ng
23 X Nitrophenol ng
24 X Nitrophenol + KO ng
25 – Thymol KO – ng
26 Lactic acid KO ng
27 Picric ammon – ng
28 Cresylic acid KO ng

30 X Succinic a KO ng

~~30~~ 29 – Succinic acid – Con

31 Balsam Copaiba + KO ng

32 ~~Lactose ng~~

33 X " KO – KO dissolves some

34 X ammon sulphophenyl ng

35 – glycerin ng

36 " + KO — KO dis some

37 Succinamid – KO ng

38 Nitroso B-Napthol KO ng

39 Sulpho cinnic acid ng

40 – Sulpho " " + KO ng

41 – Sulphocarbamid – ng blues molybdic sal —

42 " " + KO – Glucose ng

43 Nitrobenzaldehyde + KO ng

44 pyrocatechin ng

46 X Molybdate ammon + free acid ng
47 Albumen + KO
48 gum Dextrin + KO + KO dis. sam.
49 meta Dinitrobenzol + KO ng
50 ammonia Glycerbizzin ng
51 " " + KO ng

156 – Treated several times with Sulphuric Acid both strong & weak & once with Citrate Ammonia – Last time heated with strong SO_4 & got nearly a pin head of yellow pp – I sent this to B & B to see what amount of P is left when Acids take nothing from ore – .005 B & B June 9 –

157. treated 24 hours 1.4 cc Sul acid Sp gr 1.85 – 890 cc water stirred except at night. when finally washed put little of the 890 fresh Sal in to make it acid for a moment only for fear there was no free acid. Hot plate

[ITEM FOUND IN BOOK]

C O P Y.

BOOTH, GARRETT & BLAIR,
Analytical Chemists,

406 Locust Street,
Philadelphia, Pa. June 16th, 1892.

N. J. & Pa. Concentrating Works,

Dear Sirs:-

The two samples No's 157 and 158, of Iron Concentrates, which we received from you on the 14th inst, contain:

No. 157, Phosphorus,-------- 0.021%

No. 158, Phosphorus,-------- 0.026%

Sample No. 156 we will send you as soon as we can repeat our analysis, an accident having happened to our first determination.

(Signed) Booth, Garrett & Blair.

[ITEM FOUND IN BOOK]

BOOTH, GARRETT & BLAIR.
ANALYTICAL CHEMISTS.

406 Locust Street.
Philadelphia, June 18th 1892

N. Jersey & Pennsylvania Concentg. Wks.

Edison Building Broad St. N.Y.

Gentlemen:-

The sample of finely ground ore received from you a few days since marked "156" contained as received and not dried:

Phosphorus..... 0.005%

Yours respectfully,

Booth Garrett & Blair

RECEIVED
JUN 21 1892
Ans'd _____ 18

157 = As it took several hours on hot plate before metallic iron all dissolved it looks as if the ore should be 100 mesh and left one week on hot plate, or better continuously percolating apparatus — using 1000 cc water, 1 cc SO_4 — .021 Bl+B

If this sample shows less than .015 then I believe it can be brought to .010 in 3 or 4 days with above solution =

10	225°	48
11	"	72
12	"	19
13	11	01

158. 800 cc water. 1 cc
1·8 SO_4 — on bath 100 hours
twas acid up to 70 hours
in washing poured out all then
added waters & little acid &
washed it. $\frac{1}{3}$ of it dont
seem to show the usual granular
of yellow salt. = twas on hot plate
·026 Alg 1B

I had a 1000 cc. $1\frac{1}{2}$ cc Ammonia
no iron comes out first day.
a test of small granular
shows 2/3 of what SO_4 does
but after 70 hours even
when washed with SO_4
scarcely any phos —

No 1 30 grams Modoc
Arsenical py - about 150
mesh - plenty HCl - Steam
bath = ~~Heavy gold dirt~~ -
it didnt clean it much or
decompose pyrites. put
Residue on glass & very
slowly run it to Edge
shaking - under Micro -
plenty gold fine & large but
so dirty with dirt of pyrite
coned scarcely tell it was
gold - Carried hand the
Edge on watch glass
& bleached for instant with
nitric this brightened it
little but considerable
pieces were lost in form
in small glass & poby
floating I notice a great
deal way to float

Consider K strips chars
Considerably - may be that
actual contact of pyrite
dust with gold in acid
solution produced chemically the
acid & gas which lighten
Gold. I think this experiment
proves that that gold is
present but cant be seen by
Eye & very hard to see with
where & also that there is lot
of silver which reduces Spec g
to say 12 or 13. & the pyrite
lightens it & may be the gas
helps there is only just enough
Spec g diff to cause separation
of gold - If sized material were
cooled or semi roasted to lighten
or magnet used influ roast
to decrease Spec g then a
very delicate buddle or opal
fine gold could be separated
off that was fine to 80% or more of this

No 2 30 grms Modoc Arsenical
pyrite - 3/4 water 1/4 HNO3
thoroughly dissolve -

Feb. 1st 1893

Experiments made by Mr. Edison for the removal of Phosphorus from magnetic concentrates.

100 grams ring Ogden ore sieved through a 50 mesh brass wire screen to get lumps out, then washed (not near perfectly) and put in bottle with 35 cc of a 2 % solution of water and commercial H_2SO_4, shook by Harris at times for two weeks.

Liquid poured out in filter, then bottle filled again and this poured on filter, liquid was slightly acid, added molybdate solution, got considerable precipitate, then washed ore 3 times with water, and then left say 70 cc water in ore and added 10 cc of a 2 % solution H_2SO_4, and shook for 5 minutes. Poured liquid on filter, half filled bottle with water to wash out well, and poured this on filter. This solution also gave good ppt. of

(over)

on Feb 20 poured out 3 pounds of Ammonium sulphide - ~~[illegible]~~ added say 20 cc water to wash it all out of jar. Heat ore phosphorus it then washed the ore with water - then added 10 cc of 2% sol Sec acid shook & allowed it stand 10 minutes found about 3 or more phos as in the ammonium sul solution came had

Reported to Mr Edison on Monday Feb 6th

Dr Nichols been absent

Tomorrow N H Sulphide and to better clean with alcohol after acid - combine over 2nd & send to B 294 B

Feb 27 for phos assay

000027

yellow salt, showing that the Phosphoric acid recombined with iron or alumina, etc, and required an acid solution to remove it.

Washed the again 3 times, and now added 4½ cc of a 2% sol of sulphide of Ammonia, $[(NH_4)_2S]$, with 15½ cc H_2O, of course there is already say 10 or 12 cc of water in net ore.

Put $(NH_4)_2S$ in on Monday Feb. 13th 1893

On Feb 20 poured out sol. of $(NH_4)_2S$, added say 20 cc H_2O to wash it all out, found great deal of phos. in it. Then washed the ore well with water and then added 10 cc. of 2% sol. H_2SO_4, shook, allowed to stand 10 minutes, found about 1/3 as much Phos as in $(NH_4)_2S$ solution, showing some had reverted to phos. Fe or Al, or had not been attacked. Possibly $(NH_4)_2S$ (ous) would do better alone

with slight after acid.
Washed out, dried, and sent to
B. C + B, Feb 20th for Phos. assay

900000
.02½

450000
180000
22,500
40000
62,500
15625
46875
12000
58875

[ITEM FOUND IN BOOK]

[CA. FEBRUARY 13, 1893]

100 grammes Reg Ogden ore
sieved through a -50 mesh
brass sieve screen to get
lumps out, then washed (not
near perfectly) + put in bottle
with 35 ~~grams~~ cc of a 2%
solution of water + Commercial
Sulphuric acid - shook by
hand at times for two weeks -
Then poured out in filter, then bottle
filled again + then poured in
filter - liquid was slightly
acidulated Molybdic Sol
acid - got considerable precip - then
washed ore 3 times with water
then left say 20 cc water
in ore + added 10 cc of
2% sol of Sul acid + shook

2

+ shook for 5 minutes -
poured liquid on filter +
added bottle. 1/2 full water
again to wash out well +
poured this on filter -
This solution also gave good
precip ~~of phosph~~ yellow
salt, showing that the
phosphoric acid recombined
with iron or aluminate
+ evaporated the solution
acid to 6 oz got out =
Washed ore again 3
times + now added
4 1/2 cc of a 2% sol of sulphide
of ammonium - with 15 1/2 cc
of water of course there is
say 60 or 12 cc of water already
present Sul NH - Monday Feby
13 1893

[ITEM FOUND IN BOOK]

Dally.

Make some very fine Copper by reducing Sulphate with Zinc violently agitated so it dont clot, wash well & dry Naturally, Edwin will explain Mention —

See if Jacob B8 succeeds with the formalin tartaric Compound

Mab

Dally —

Boil Morlain with Nitrate of Lead also acetate lead till lead succeeds also Bismuth acetate — none

Make a Gelatin Emulsion in water mix therewith Linseed oil & Castor oil then agitate the solution with formaldehyde & glycerine

Make Hydric acid from Sugar with weak H_2SO_4 boiled —

Make = Silicate Soda 6 parts glycerine 1 part, Red lead 3½ parts. the Silicate & Glycerine are mixed & then the Red lead — to thicken 10 parts of mixture under like band — it goes to a pulley

[ITEM FOUND IN BOOK]

602
649
653
651
635
670
671
669
672
686
640
601
604
641
614
620
603
631
656
682
737
677
678
699
719
667
731

Heat your butter dishes
so Mercurous will cool slowly

Dolley

~~Powdered Mercury, then 150 or 200 mesh put in Good Conc Nitric acid for several hours — wash free of acid & see how it melts. Save a part of the oxidized Mercury & put little acid KOH with it & melt.~~

~~Add little hunks of NaOH to Melted Mercury — try various proportions —~~

Add to Melted Mercury —
~~Manganese Sulphide —~~
also ~~Dry Potassium Sulphide~~ (yellow kind — Tin box) also precipitated pink ~~Arsenic Sulphide~~ — also ~~finally precipitated Antimony Sulphide —~~

~~Powdered Mercury 200 or 150 mesh put 3 or 4 grams to soak in small beaker with formaldehyde~~

[ITEM FOUND IN BOOK]

2

Burn some rubber ~~[illegible]~~ same as was used on glass & rap Cracks add various proportions to ~~[illegible]~~ Mellod Montan —

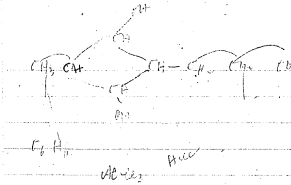
~~Add to Mellod Montan powdered Caustic Lime — also try finely slaked Lime —~~

~~Calls of some Lumsdon & Cofilms from a paint factory see if a suitable Mellod Montan~~

~~Test all the different Asphalts. Use have & try each one Each one with a different proportion~~

~~Try Acetates of the Metals you will have to keep the mix that mixed Most of the acetic acid is gone you can still by the smell —~~

Mix Oleate Alumina with Montan



Dissolve in benzole and warm in a refluxing ~~[illegible]~~ cooler and then add small quantity of Chloride Aluminum boil a long time then wash out with water and heat until free from water

[ITEM FOUND IN BOOK]

Naphtalin Bichromat.

Carbonium Trichlorat

Diphenylamine

Meta Phenylendiamine Base

Hexachloro Naphtol

Amidophenol Para.

Naphtalenin Hydrochlor.

Alumida

Cyrin asphalt

soft Mexican

Tarin pitch

Pyhne asphalt

opal

crude Trinidad

Gilsonite

Refined trinidad

Cubam

Hard Mexican

[ITEM FOUND IN BOOK]

copper sulphate
Alum - for Aluminum sulphate
Magnesium sulphate - Epson salts
Manganese sulphate
zinc Sulphate
Iron Sulphate
Lead Nitrate

[ITEM FOUND IN BOOK]

Iron Protochloride
Stearate
ferric Sulphate (Basic
Iron sulphate
ferrous Citrate
Mang. lacate
Amm Protosulphate
Perchloride
Resinate
acetate
Ferrick Amm Tartras
" Citras
Tartrate
Iron Carbonate
Fer Oxydat Hydrat
Ferret Potass Tart
ferrum Tannic
Iron phosphate
Amm persulph.
Chloride
Ferret Potass Tart
Iron Protosulphate

Persulphates
perioides
Chlorates
Perchlorates
Phosphorus
Hypo chlorates
Bromates

[ITEM FOUND IN BOOK]

Barium Carb 701
Cadmium " 702
Lith " " 703
Iron " " 704

696
24
720

[ITEM FOUND IN BOOK]

We find that in the nine samples of concentrates received from you May 2 marked as below

	Phosphorus	Sulphur
#137 –	0.016 –	0.069
#138 –	0.015	
#139 –	0.017	
#144 –	0.014	
#145 –	0.015 –	0.084
#148 –	0.017	
#149 –	0.014	
#150 –	0.023	
#152 –	0.022	

Robt Gowen & Blair